

AIR RESOURCES BOARD

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PROCEDURE FOR THE DETERMINATION OF DISTILLATION POINTS OF
LIQUID FUELS BY AUTOMATED DISTILLATION

SOP NO. MLD 128

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CALIFORNIA AIR RESOURCES BOARD
MONITORING AND LABORATORY DIVISION

Standard Operating Procedure for the Determination of Distillation
Temperatures of Liquid Fuels by Automated Distillation

1 Introduction

- 1.1 This document describes the standard operating procedure (SOP) for measuring the distillation temperatures of liquid fuels using an automated distillation instrument.
- 1.2 This SOP covers gasoline, diesel fuel, aviation fuel, kerosene, and similar petroleum products.
- 1.3 This SOP is based on ASTM D86-99^a¹.

2 Method

- 2.1 A 100 mL sample is introduced into the instrument's flask.
- 2.2 The sample is distilled under specific conditions depending on its characterization.
- 2.3 Vapor temperature and condensate volume are periodically measured. These data are used to calculate results.

3 Instrumentation

- 3.1 Herzog Automated Distillation unit, model MP626, MP627, or MP628.
- 3.2 Data acquisition system: Herzog MP626/7/8 software running under Microsoft Windows.
- 3.3 125 mL distillation flask
- 3.4 100 mL graduated cylinder appropriate for use with 3.1 above

4 **Reagents**

- 4.1 Toluene, A.C.S. reagent grade or better.

5 **Preparation of Instrument**

- 5.1 The MP626 should be turned on at least 30 minutes before analyses are performed. Later versions of the instrument do not require any warm-up.
- 5.2 The correct distillation method is selected within the software. Method D86-123-1 is used for gasolines with Reid vapor pressure (RVP) greater than 9.5. Method D86-123-2 is used for gasolines with RVP less than 9.5.
- 5.3 The initial heater temperatures, switching time, and final heat adjustment time are selected by the operator based on the sample RVP and any previous distillation analyses performed (see 7.11).

6 **Calibration**

- 6.1 Two systems of the Herzog MP626/7/8 require calibration: temperature measurement and volume measurement.
- 6.2 Temperature calibration is performed by distilling pure toluene on the instrument and comparing the result with the historical reference value of 228.2 °F. Any observed difference between the two readings is used by the software to calibrate the instrument's temperature sensor.
- 6.3 Temperature calibration is carried out at least once every six months or whenever the temperature sensor is changed.
- 6.4 Volume calibration is performed by inserting an empty volumetric flask into the MP626/7/8 receiver area and selecting "Zero point adjust" from the software.
- 6.5 Volume calibration is carried out at least once every six months or whenever the volumetric flask is changed.

7 Procedure

- 7.1 A piece of soft, lint-free cloth attached to a metal wire is used to clean the instrument's condenser tube. It is inserted into the receiver tube and pulled gently through the distilling compartment.
- 7.2 The sample is chilled to a temperature between 32 °F and 50 °F.
- 7.3 The graduated cylinder and distillation flask are chilled to a temperature between 55 °F and 65 °F.
- 7.4 The sample is poured into the graduated cylinder. The bottom of the meniscus should line up with the 100 mL mark.
- 7.5 The sample is transferred as completely as possible from the graduated cylinder to the distillation flask.
- 7.6 The distillation flask is centered on the MP626/7/8 heating plate with the side arm connected to the inlet of the condenser tube.
- 7.7 The temperature sensor is inserted into the distillation flask. The sensor's measuring element must be exactly level with the lower inside edge of the flask's side arm.
- 7.8 A metallic drop plate is inserted into the top of the graduated cylinder. The cylinder is inserted under the outlet of the condenser tube. A piece of insulating material cut to fit the cylinder is used as a cover.
- 7.9 The distillation is started and monitored via the instrument software.
- 7.10 The following parameters must be met for a distillation to be considered valid:
 - 7.10.1 Time to first drop: 5 to 10 minutes for gasoline, 5 to 15 minutes for heavier products.
 - 7.10.2 Time from first drop to 5% recovered: 60 to 100 seconds for gasoline, not applicable for heavier products.

7.10.3 Distillation rate: 4 to 5 mL/min

7.10.4 Time from final heat adjustment to end point: 0 to 5 minutes

8 **Quality control**

- 8.1 According to ASTM D86-99^{ae1}, if any of the parameters in 7.10 is not met, the distillation run is not considered valid. However, in practice, failure to meet all these criteria does not make a significant difference in the data obtained. The acceptability of data obtained from an invalid run is determined by the client.
- 8.2 In order to obtain data which can meet ASTM D86-99^{ae1} requirements, repeated runs with different initial temperatures, switching times, heater curve modifications, and final heat adjustment volumes are used.
- 8.3 At least once every six months, the distillation temperature of pure toluene is determined and compared with the historical reference value of 228.2 °F. Any difference between the two values is used by the instrument software to correct the automated instrument's reading.

9 **References**

- 1. "Standard Test Method for Distillation of Petroleum Products (Designation D86-99^{ae1})," *Annual Book of ASTM Standards*, Vol 05.01.
- 2. "User Manual for MP-626," Walter Herzog GmbH, Lauda-Königshöfen, Germany, 1995.
- 3. "User Manual for MP-627/8," Walter Herzog GmbH, Lauda-Königshöfen, Germany, 1996.
- 4. "User Manual for Software HLIS," Walter Herzog GmbH, Lauda-Königshöfen, Germany, 1995.

10. **Standard Operating Procedure Revision History**

- Version 1.1: Adopted 10/1/97.
- Version 1.2: Adopted 4/1/00. Section 8.3 modified to include mesitylene distillation, which had been inadvertently omitted.
- Version 1.3 Adopted 7/1/01. All references to MP 626 changed to MP 626/7/8 to reflect the presence of different models of the instrument. References to ASTM method updated to D86-99^{ae1} to reflect regulatory changes. Requirements in section 7.10 updated to reflect the new version of D86. Calibration frequency changed to once per six months to mirror the requirements of D86.
- Version 1.4 Adopted 4/1/02. References to mesitylene distillation removed and all references to manual distillation removed. Section 5.1 updated to reflect newer instrument models. Section 7.1 changed to reflect current practice, as advised by the instrument manufacturer.
- Version 1.41 Adopted 10/1/02. Section 7.10 clarified to distinguish between gasoline and heavier products. MP627 manual reference added.